

SVOJSTVA KEMIJSKI AKTIVIRANE UGLJIČNE ANODNE PRAŠINE PROPERTIES OF THE CHEMICALLY ACTIVATED CARBON ANODE DUST

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Sažetak: Ugljična anodna prašina (UAP) otpadni je materijal pri proizvodnji aluminija. Kemijski sastav UAP određen je metodom apsorpcijske spektrometrije (AAS), a mineraloški sastav metodom difrakcije rendgenskog zračenja (XRD). Površinska svojstva (specifična površina, prosječni promjer pora i ukupni volumen pora) određeni su Brunauer-Emmett-Telleorovom (BET) metodom i Barrett-Joyner-Halendainom (BJH) metodom. Mikroskopska ispitivanja provedena su pretražnom elektronском mikroskopijom (SEM). Sva su ispitivanja provedena prije i nakon kemijske aktivacije UAP s KOH, ZnCl₂ i organskim kiselinama (octenom i limunskom). Dobiveni rezultati pokazali su pozitivan utjecaj kemijske aktivacije na kemijska i strukturalna svojstva ugljične anodne prašine kao potencijalnoga jeftinog adsorbensa.

Ključne riječi:

- ugljična anodna prašina
- otpadni materijal
- kemijska aktivacija
- kemijska i fizikalna svojstva

Abstract: The carbon anode dust (CAD) is waste material of the aluminium production industry. The chemical and mineralogical composition of CAD was examined by atomic absorption spectroscopy (AAS) and the X-ray diffraction method (XRD), respectively. Surface area properties (specific surface area, average pore diameter, total pore volume) are determined by the Brunauer-Emmett-Teller (BET) and Barrett-Joyner-Halenda (BJH) methods. Microscopic observation was conducted using scanning electron microscopy (SEM method). All of investigations were performed before and after chemical activation of CAD by KOH, ZnCl₂ and by organic acids (acetic and citric). The results obtained suggest the positive effect of chemical activation on the chemical and structural properties of carbon anode dust as a potentially low-cost adsorbent.

Keywords:

- carbon anode dust
- waste material,
- chemical activation,
- chemical and structural properties

1. UVOD

Hall-Heroultovim postupkom aluminij se proizvodi elektrolitičkom redukcijom glinice rastaljene u elektrolitu koji sadrži kriolit kao sredstvo za taljenje. Za redukciju se upotrebljavaju ugljične anode. Dijelovi nepotrošenih anoda iz postupka proizvodnje aluminija nazivaju se anodnim ostatkom. Postoje dvije vrste anodnog ostatka: sirovi i pečeni. Očišćeni anodni ostaci melju se i ponovo upotrebljavaju u proizvodnji novih anoda (oko 20 % anode reciklira). Ugljična anodna prašina nastaje za vrijeme pečenja i transporta anoda i ne reciklira se. Zbog svojega granulometrijskog i kemijskog sastava (mogućih nečistoća) ugljična anodna prašina industrijski je otpadni materijal [1].

1. INTRODUCTION

In the Hall-Heroult process, aluminum is produced by the electrolytic reduction of alumina dissolved in an electrolyte containing cryolite as a flux agent. For reduction, carbon anode has been used. The remaining parts of spent anodes from the aluminium production are called anode butts. There are two anode butts, the raw anode butts and the pre-baked anode butts. The cleaned anode butts are crushed and reused in the production of new anodes (about 20 % of the anode is recycled). Carbon anode dust (CAD) originates during the baking and transport of anodes and it does not recycle. Because of their granulometry and chemical composition (possible impurities), carbon anode dust is an industrial waste material [1].

Aktivni ugljici su najčešće upotrebljavani adsorbensi u procesima separacije i pročišćavanja. U posljednje su vrijeme intenzivirana nastojanja znanstvenika za pronaalaženjem alternativnih adsorbensa koji bi zamijenili skupe aktivne ugljike. Industrijski otpadni materijali jedni su od potencijalno jeftinih adsorbensa za uklanjanje metala i organskih tvari.

Svrha aktivacije je dobivanje učinkovitih jeftinih adsorbensa. Aktivacija se može provesti kemijskim i fizikalnim postupcima. Pri kemijskoj aktivaciji osnovni se materijal impregnira raznim kemijskim sredstvima, a zatim karbonizira. Kao najčešći aktivatori poznati su H_3PO_4 , $ZnCl_2$ i spojevi alkalijskih metala [2]. Fizikalna aktivacija podrazumijeva karbonizaciju ugljične sirovine, nakon čega slijedi obrada nastalog karbonizata plinovima, ili pak direktnu aktivaciju početnog materijala aktivirajućim agensima (kao što su CO_2 , vodena para, N_2 , H_2 , O_2) [3-5].

U radu su ispitana svojstva ugljične anodne prašine prije aktivacije različitim kemijskim sredstvima i nakon nje. Svrha tih istraživanja bila je procjena mogućnosti upotrebe aktivirane UAP kao potencijalnoga jeftinog adsorbensa.

2. EKSPERIMENTALNI DIO

2.1. Uzorci

Ispitana je ugljična anodna prašina koja je čvrsti ostatak pri proizvodnji aluminija. Uzorci su sušeni 4 sata na $105^{\circ}C$ i prosijani na veličinu čestica $\leq 0,2$ mm. Kemijski sastav uzorka određen je atomskom apsorpcijском spektroskopijom (AAS metoda) spektrometrom AA-6800, Shimadzu. Mineraloški sastav ugljične anodne prašine određen je metodom difrakcije rendgenskog zračenja (XRD) upotrebom difraktometra Philips PW 1710 s CuK_{α} zračenjem.

2.2. Postupak aktivacije

Kemijska aktivacija anorganskim sredstvima (KOH i $ZnCl_2$) provedena je na sljedeći način. Četiri grama ugljične anodne prašine miješano je s otopinom koja je sadržavala 20 ml vode i 4 g sredstva za aktivaciju. Dobivena smjesa bila je u kontaktu 2 sata na $20^{\circ}C$, a zatim 2 sata na $400^{\circ}C$. Uzorci su višestruko ispirani destiliranom vodom sve dok pH-vrijednost nije poprimila početnu vrijednost. Nakon toga uzorci su sušeni 2 sata na $110^{\circ}C$. Dobivena smjesa karbonizirana je u peći s inertnom atmosferom. Uzorci su zagrijavani 1,5 sati od sobne temperature do temperature zagrijavanja ($700^{\circ}C$). Brzina zagrijavanja bila je $10^{\circ}C/min$ u struji argona protoka 5 l/h. Zatim su dobiveni uzoci ohlađeni na sobnu temperaturu i

Activated carbons are the most widely used adsorbents in separation and purification processes. Research interest into the production of alternative adsorbents to replace the costly activated carbons has been intensified in recent years. Industrial waste is one of the potentially low-cost adsorbent materials for metal and organic matter removal.

The purpose of activation is the production of effective but not expensive adsorbents. Activation can be carried out by chemical or physical means. In chemical activation, the precursor is impregnated with a different chemical agent and, after that, it is carbonized. The most commonly used activating agents have been reported as H_3PO_4 , $ZnCl_2$ and alkaline metal compounds [2]. Physical activation involves carbonization of a carbonaceous precursor followed by gasification of the resulting char or direct activation of the starting material in the presence of an activating agent (such CO_2 , steam, N_2 , H_2 , O_2) [3-5]. In this work, properties of carbon anode dust before and after chemical activation by different agents are examined. The aim of these investigations was to evaluate the possibility of the CAD activation in order to produce a potentially low-cost adsorbent.

2. EXPERIMENTAL

2.1 Samples

The carbon anode dust, which is a solid residue of aluminium production, was investigated. The samples were dried at $105^{\circ}C$ for 4 hours and sieved to a particle size $\leq 0,2$ mm. The chemical composition of the ground sample was determined by atomic absorption spectroscopy (AAS method) on AA-6800, Shimadzu spectrometer. Mineralogical composition of an anode dust sample was determined by the X-ray diffraction method (XRD method) using a Philips PW 1710 diffractometer with CuK_{α} radiation.

2.2 Activation procedure

Chemical activation by inorganic agents (KOH and $ZnCl_2$) was carried out using following procedure: Four grams of carbon anode dust were mixed with a solution that contained 20 ml of water and 4 g of the activation agent. The obtained mixtures were in contact for 2 hours at $20^{\circ}C$ and then for 2 hours at $400^{\circ}C$. Subsequently the samples were washed repeatedly with distilled water until the initial value of solution pH was established. Afterwards, the samples were dried for 2 hours at $110^{\circ}C$. The resulting mixture was carbonized in a furnace under argon flow. The samples were heated for 1,5 hours from room temperature to the final heating temperature ($700^{\circ}C$). The heating rate was $10^{\circ}C/min$ under an argon

isprani destiliranom vodom radi uklanjanja ostatka kemikalija.

Kemijska aktivacija organskim kiselinama provedena je na sljedeći način. Ugljična anodna prašina (6,4 g) miješana je tresilicom 30 minuta s 40 ml kiseline koncentracije 1 mol/l pri 20 °C. Dobivena suspenzija filtrirana je kroz filter-papir Whatman No. 40 kako bi se izdvojila ugljična anodna prašina iz otopine. Sljedeći korak bio je ispiranje destiliranom vodom i sušenje dobivenog uzorka na 90 °C.

Sve upotrijebljene kemikalije bile su analitičke čistoće.

2.3 Analiza površinskih svojstava

Površinska svojstva određena su Brunauer-Emmett-Tellerovom (BET) metodom. Za određivanje specifične površine i ukupnog volumena pora određene su izoterme adsorpcije-desorpcije N₂ na ispitanim uzorku upotreboom uređaja Micromeritics ASAP 2000. Prosječni volumen pora izračunat je iz izraza:

$$d = \frac{4 \cdot V_p}{S_p} \quad (1)$$

gdje su V_p-specifični volumen, a S_p-specifična površina. Raspodjela veličina pora izračunata je Barrett-Joyner-Halendinom (BJH) metodom.

2.4 Ispitivanje morfologije površine

Morfologija površine uzorka UAP ispitana je metodom kvalitativne pretražne elektronske mikroskopije (SEM) upotrebom mikroskopa Jeol JXA 50A.

3. REZULTATI I DISKUSIJA

Rezultati kemijske analize pokazali su da u UAP najviše ima ugljika ((92 %). Slijede Si (1,73 %), Al (1,69 %), S (1,5 %), Fe (0,34 %) itd. Prema rezultatima dobivenima primjenom XRD-metode, grafit, α-kvarc, mulit i α-kristobalit glavne su identificirane mineraloške komponente.

Adsorpcijska sposobnost adsorbensa ovisi o kemijskom i mineraloškom sastavu te strukturnim karakteristikama. Podaci koje donosi literatura pokazuju da bi se UAP s obzirom na kemijski i mineraloški sastav mogla smatrati djelotvornim adsorbensom [2,6]. To se odnosi na njezin visok sadržaj ugljika i mineraloški sastav (oksidi i

flow of 5 l/h. The obtained products were then cooled to room temperature and washed with distilled water to remove remaining chemicals.

Chemical activation with organic acids (acetic and citric) was carried out using following procedure: The carbon anode dust (6,4 g) was mixed with 40 ml of an acid solution concentration of 1 mol/l at 20 °C by means of an mechanical shaker (for 30 minutes). The resulting suspension was filtered through a Whatman filterpapir No.40 to separate the carbon anode dust from the solution. The next step was washing with distilled water and drying of the obtained samples at 90 °C. All chemicals used in the study were of an analytical grade.

2.3 Analysis of surface area properties

The surface area properties were determined by the Brunauer-Emmett-Teller (BET) method. The N₂ adsorption-desorption isotherms of the investigated samples were measured using a Micromeritics ASAP 2000 adsorption instrument in order to determine the surface area and total pore volume. The average pore diameter is calculated using the equation

$$d = \frac{4 \cdot V_p}{S_p} \quad (1)$$

where V_p is specific volume and S_p is the specific surface area. Pore size distribution of CAD is calculated by the Barrett-Joyner-Halenda (BJH) method.

2.4 Examination of surface morphology

Surface morphology of CAD samples was examined by the method of qualitative scanning electron microscopy (SEM) using a Jeol JXA 50A microscope.

3. RESULTS AND DISCUSSION

The results of chemical analysis have shown that CAD is dominated by C (92 %). Then follow Si (1,73 %), Al (1,69 %), S (1,5 %), Fe (0,34 %) etc. According to the results obtained by XRD-method, graphite, α-quartz, mullite and α-cristobalite are the major identified mineralogical components.

The adsorption capability of an adsorbent depends on chemical and mineralogical composition and structural characteristics. Literature indicates that CAD with its chemical and mineralogical composition could be considered as an effective adsorbent [2, 6]. This can be related to its high carbon content and mineralogical composition (oxides and aluminosilicates).

alumosilikati). Takav sastav UAP može se objasniti procesnim uvjetima elektrolitičke redukcije glinice. Naime ugljične anode sastoje se od naftnog koksa kao punila i katranske smole kao veziva. U elektrolizi aluminija elektrolit je otopina glinice, Al_2O_3 u kriolitu, Na_3AlF_6 . Prisutnost kriolita potrebna je za otapanje glinice i da bi se omogućile niže temperature procesa [7].

Such composition of CAD can be explained by the processing conditions of the electrolytic reduction of alumina. The carbon anode is composed of the petroleum coke as a filler and coal-tar pitch as binder. The electrolyte for aluminium electrowinning is basically a solution of alumina Al_2O_3 in cryolite, Na_3AlF_6 . The presence of cryolite is essential for dissolution of alumina as well as for lower operating temperature [7].

Tablica 1. Površinska svojstva ugljične anodne prašine prije aktivacije i nakon nje

Table 1. The surface area properties of carbon anode dust before and after the chemical activation

| SVOJSTVA PROPERTIES | PRIJE AKTIVACIJE BEFORE ACTIVATION | NAKON AKTIVACIJE AFTER ACTIVATION | | | |
|--|---|--------------------------------------|----------------------|----------------------------------|--------------------------------|
| | | KOH | ZnCl_2 | Limunska kiselina Citric acid | Octena kiselina Acetic acid |
| Specifična površina, Surface area, $S_{\text{BET}}, \text{m}^2/\text{g}$ | 6,48 | 5,87 | 7,12 | 7,82 | 7,69 |
| Volumen pora od 1,7 do 300 nm, Pore volume from 1,7 to 300 nm, $V, \text{cm}^3/\text{g}$ | $2,37 \cdot 10^{-2}$ | $1,95 \cdot 10^{-2}$ | $1,87 \cdot 10^{-2}$ | $1,55 \cdot 10^{-2}$ | $1,88 \cdot 10^{-2}$ |
| Prosječni promjer pora, Average pore diameter, d, nm | 13,22 | 11,45 | 8,96 | 7,45 | 9,49 |

U tablici 1 prikazani su rezultati površinskih svojstava UAP: specifična površina, prosječni promjer pora i ukupni volumen pora (1,7-300 nm), prije aktivacije i nakon nje. Nakon aktivacije s ZnCl_2 i limunskom kiselinom specifična površina neznatno raste te iznosi $S_B = 7,12 \text{ m}^2/\text{g}$ odnosno $S_B = 7,69 \text{ m}^2/\text{g}$.

Prosječni promjer pora aktiviranih uzoraka manji je u odnosu na vrijednost prije aktivacije UAP ($d=13,2 \text{ nm}$). Prema IUPAC klasifikaciji, ugljična anodna prašina je mezoporozan materijal ($2 \text{ nm} < d < 50 \text{ nm}$). Vrijednosti za ukupni volumen pora (1,7-300 nm) kemijski aktivirane UAP niže su od ukupnog volumena pora neaktivirane UAP ($V = 2,37 \cdot 10^{-2} \text{ cm}^3 \text{ g}^{-1}$).

Slika 1 prikazuje ukupnu raspodjelu veličina pora, $Q_3(d)$ za sve ispitane uzorke. Vidljivo je da je aktivacijski proces izmjenio raspodjelu veličina pora tj. udio malih pora raste. Te rezultate nadopunjaju ispitivanja morfologije površine. Na sličici 2 je SEM izgled čestica UAP prije aktivacije. Vidljive su heterogenosti i pore različite veličine (sličica 2a). Slična mikroskopska opažanja dobivena su za uzorak UAP aktiviran s KOH (sličica 2b). Obje kiseline, octena i limunska, reducirale su broj pukotina na površini UAP (slike 2c i 2d). Aktivacija limunskom kiselinom više je

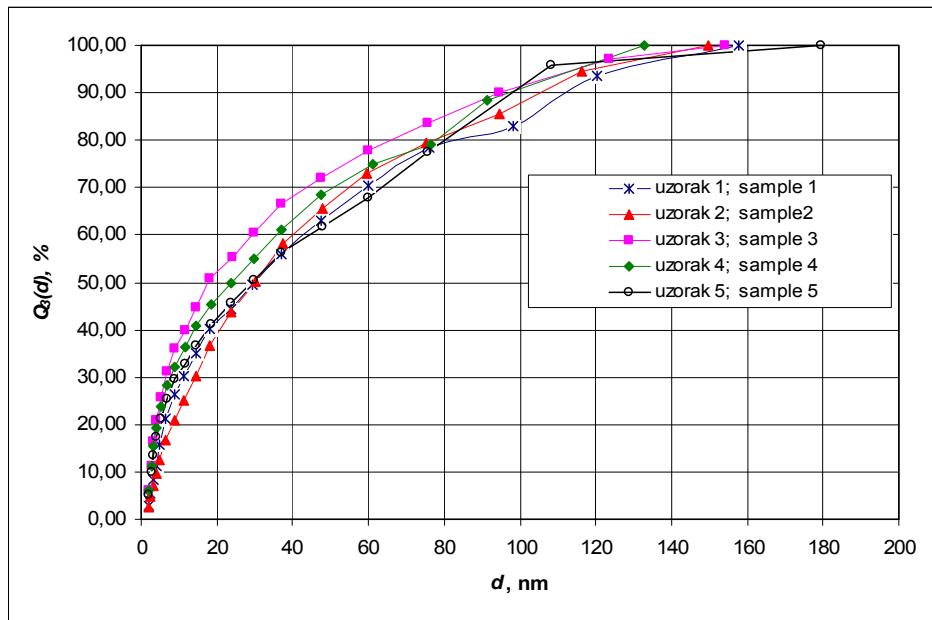
Table 1 presents the results of the surface area properties of CAD: specific surface area, average pore diameter, total pore volume (1,7-300 nm) before and after activation. The value of BET surface area for nonactivated CAD is $S_B = 6,48 \text{ m}^2/\text{g}$. The BET surface area slightly increased after activation by ZnCl_2 ($S_B = 7,12 \text{ m}^2/\text{g}$), and citric acid ($S_B = 7,69 \text{ m}^2/\text{g}$) respectively.

The average pore diameter of the activated samples is decreased in relation to the value of 13,22 nm for the CAD sample before activation. According to IUPAC classification, carbon anode dust is a mesoporous material ($2 \text{ nm} < d < 50 \text{ nm}$). The values of total pore volume (1,7-300 nm) of chemically activated CAD are lower than total pore volume for nonactivated CAD ($V = 2,37 \cdot 10^{-2} \text{ cm}^3 \text{ g}^{-1}$). Figure 1 shows cumulative pore size distribution ($Q_3(d, \%)$) for all investigated samples. It can be seen that the activation process changed the cumulative pore size distribution i.e. the fraction of the small pores increased. These results were confirmed by surface morphology investigations. Figure 2 shows the SEM image of a carbon anode dust sample before chemical activation. Some heterogeneities and pores of different size (Figure 2a) are visible.

A similar microscopic observation is given for a CAD

promijenila izgled površine UAP nego aktivacija octenom kiselinom. Štoviše, slika 2c prikazuje glatku ravnu površinu nakon aktivacije UAP limunskom kiselinom.

sample activated by KOH (Figure 2b). Both acetic and citric acids reduced the number of cracks on the CAD surface (Figures 2c and 2d). Activation by citric acid has changed the image of the CAD surface more than the acetic acid. Moreover, Figure 2c displays a smooth flat surface after CAD activation by citric acid.

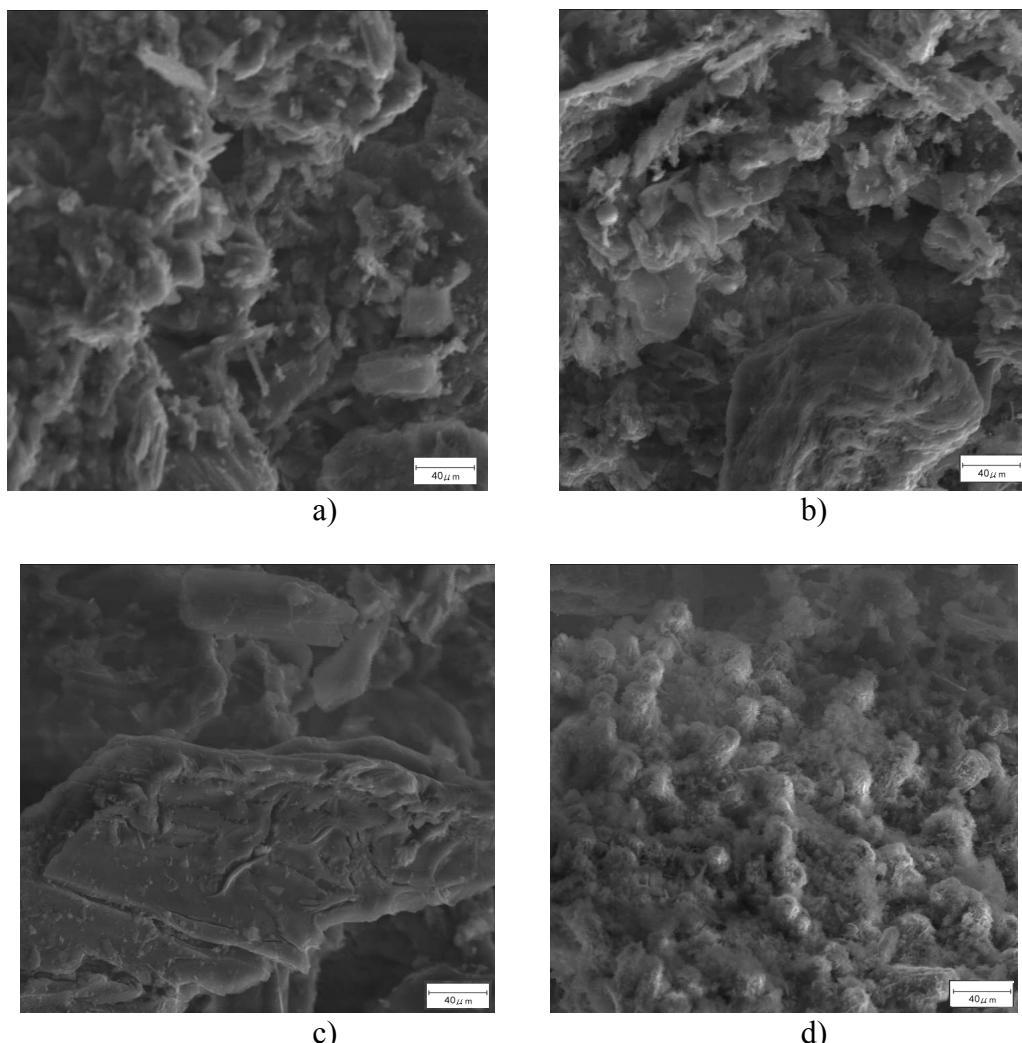


Slika 1. Ukupna raspodjela veličine pora ispitivanih uzoraka, $Q_3(d)$, %: neaktivirani (uzorak 2), uzorak aktiviran s KOH (uzorak 1), limunskom kiselinom (uzorak 3), octenom kiselinom (uzorak 4) i $ZnCl_2$ (uzorak 5)

Figure 1. Cumulative pore size distribution of, $Q_3(d)$, % investigated CAD sample: nonactivated (sample 2) and activated by KOH (sample 1), citric acid (sample 3), acetic acid (sample 4) and $ZnCl_2$ (sample 5)

Iako je kemijska aktivacija upotrebljavana godinama, njezin mehanizam nije jasan. Opće je prihvaćeno djelovanje aktivirajuće tvari kao dehydratacijskog sredstva koje može mijenjati ponašanje ugljične tvari tijekom pirolize. Dva su mehanizma kemijske aktivacije: prvi obuhvaća nastajanje mikropora koje počinje dodatkom kemijskih tvari početnom materijalu; drugi predstavlja razvoj pora kao rezultat kemijskog utjecaja unutar otvorenih pora [4, 8].

Although a chemical activation method has been used for many years, the mechanism is not clear. It is generally accepted is that the activating agent acts to dehydrate chemicals, which may alter the pyrolysis behaviors of carbonaceous materials. In chemical activation, there are two mechanisms: the first one is the micropore formation which starts with the addition of the chemical to the raw material and the second one is the pore widening which is the result of the chemical effect inside the opened pores [4, 8].



Slika 2. Izgled čestica ugljične anodne prašine snimljen pretražnim elektronskim mikroskopom (SEM): a) neaktivirane i aktivirane s b) KOH, c) limunskom kiselinom, d) ZnCl₂

Figure 2. SEM image of carbon anode dust particles: a) nonactivated and activated by b) KOH, c) citric acid, d) acetic acid

Dobiveni rezultati u skladu su s podacima u literaturi. Naime, za fizikalnu aktivaciju važni su veličina i volumen pora dok za kemijsku aktivaciju važnu ulogu ima kemija površine adsorbenta (tj. različite funkcionalne grupe) [9, 10].

3. ZAKLJUČAK

Ispitivanja kemijskog i mineraloškog sastava ugljične anodne prašine pokazala su prisutnost visokog sadržaja ugljika (grafit), oksida i alumosilikata. Kemijskom aktivacijom ugljične anodne prašine (s KOH, ZnCl₂, octenom i limunskom kiselinom) promijenjena su površinska svojstva i morfologija UAP. Nakon postupka aktivacije na uzorcima UAP dolazi do povećanja specifične površine, smanjenja veličine pora i bolje ukupne raspodjele veličine pora. Mikroskopska ispitivanja pokazala su da limunska

The obtained results correspond to the literature date. Namely, in physical activation the size and volumes of pores are important whereas in chemical activation surface chemistry (i.e. different functional groups) of adsorbent plays a significant role [9, 10].

3. CONCLUSION

The examined chemical and mineralogical composition of carbon anode dust has shown high content of carbon (graphite) and the presence of oxides and alumosilicates. Chemical activation of carbon anode dust (by KOH, ZnCl₂, acetic and citric acid) has changed CAD surface area properties and surface morphology. The increasing of specific surface area, decreasing of pore size and better cumulative pore size distribution in CAD samples were registered following the applied activation procedure. The microscopic

kiselina ima znatan utjecaj na morfologiju površine ugljične nodne prašine. Zbog kemijskih i površinskih svojstava UAP bi se mogla koristiti kao porozan adsorbens. Za potpunije vrednovanje adsorpcijske sposobnosti preporučuje se određivanje kemije površine ugljične anodne prašine

observations have shown the significant influence of citric acid activation on CAD surface morphology. Owing to their chemical and surface properties, CAD could be used as porous adsorbent. For more complete evaluation of CAD adsorption capability, surface chemistry determination is recommended.

4. POPIS OZNAKA

| | | |
|-------------------------------------|----------------------------|--------------------------------|
| Ugljična anodna prašina | <i>UAP</i> | Carbon anode dust |
| Brunauer-Emmett-Teller | <i>BET</i> | Brunauer-Emmett-Teller |
| Barrett-Joyner-Halenda | <i>BJH</i> | Barrett-Joyner-Halenda |
| Pretražna elektronska mikroskopija | <i>SEM</i> | scanning electron microscopy |
| Atomska apsorpcijska spektroskopija | <i>AAS</i> | atomic absorption spectroscopy |
| Difrakcija X-zraka | <i>XRD</i> | X-ray diffraction |
| BET površina | $S_B, \text{m}^2/\text{g}$ | BET surface area |
| Specifična površina | $Sp, \text{m}^2/\text{g}$ | specific surface area |
| Specifični volumen | Vp, cm^3 | specific volume |
| Prosječni promjer pora | d, nm | average pore diameter |

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4. LIST OF SYMBOLS

| | |
|--------------------------------|----------------------------|
| Carbon anode dust | <i>UAP</i> |
| Brunauer-Emmett-Teller | <i>BET</i> |
| Barrett-Joyner-Halenda | <i>BJH</i> |
| scanning electron microscopy | <i>SEM</i> |
| atomic absorption spectroscopy | <i>AAS</i> |
| X-ray diffraction | <i>XRD</i> |
| BET surface area | $S_B, \text{m}^2/\text{g}$ |
| specific surface area | $Sp, \text{m}^2/\text{g}$ |
| specific volume | Vp, cm^3 |
| average pore diameter | d, nm |

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